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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 7
For Quarter Ending January 15, 1967

By

R. W. HARRISON
and
E. E. HOFFMAN

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-6474

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT 7

Covering the Period
October 15, 1967 to January 15, 1967

Edited by
R. W. Harrison
Project Metallurgist

Approved by
E. E. Hoffman
Manager, Corrosion Technology

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-6474

January 28, 1967

Technical Management
NASA-Lewis Research Center
Space Power Systems Division
R. L. Davies

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. For this program, Mr. R. L. Davies is the NASA Project Manager.

The program is being administered for the General Electric Company by Dr. J. W. Semmel, Jr., and E. E. Hoffman is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities procurement and test operations. R. W. Harrison, the Project Metallurgist, is responsible for the materials procurement, utilization and evaluation aspects of the program. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand, L. E. Dotson and H. Bradley.

Welding and Joining - W. R. Young and S. R. Thompson.

Refractory Alloy Procurement - R. G. Frank and L. B. Engel, Jr.

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from October 15, 1966 to January 15, 1967, of a program to fabricate, operate for 10,000 hours, and evaluate a potassium corrosion test loop constructed of T-111 (Ta-8W-2Hf) alloy. Materials for evaluation in the turbine simulator include Mo-TZC and Cb-132M. The loop design will be similar to the Prototype Loop; a two-phase, forced convection, potassium corrosion test loop which has been developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment to the water cooled vacuum chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F

b. Superheat temperature, 2150°F

c. Condensing temperature, 1400°F

d. Subcooling temperature, 1000°F

e. Mass flow rate, 40 lb/hr

f. Boiler exit vapor velocity, 50 ft/sec

g. Average heat flux in plug (018 inches), 240,000 BTU/hr ft^2

h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft^2

II. SUMMARY

During the seventh quarter of the program, work proceeded on the topics abstracted below:

The T-111 alloy tubing was received and quality assurance inspection is in progress. Most of the T-111 alloy materials have been released for the manufacture of critical components; the remaining necessary materials are being inspected.

Fabrication and installation of the lithium still into the purification system was completed and outgassing is in progress.

Fabrication of many of the loop components is in progress.

III. PROGRAM STATUS

A. MATERIALS PROCUREMENT

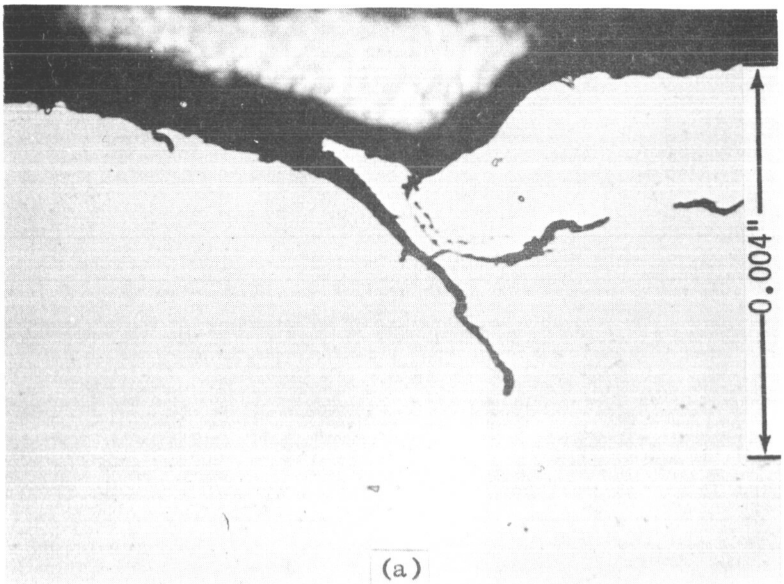
1. T-111 Alloy

The final vendor inspection and testing of the T-111 alloy 0.375-inch OD x 0.065-inch thick wall and 1.0-inch OD x 0.100-inch thick wall tubing has been completed. The tubing was received on November 18, 1966 and certified by the vendor to be free of rejectable defects. Subsequent quality assurance inspection by ultrasonic testing at General Electric revealed numerous ID and OD defects in the 0.375-inch OD tubing.

Approximately 20% of the quality assurance inspection of the tubing has been completed and numerous ID and OD defects were observed in the 0.375-inch OD tubing during ultrasonic testing by General Electric. Many of the defects were identified by visual examination, using a stereomicroscope, as OD defects of the scratch-type which were apparently introduced during handling prior to receipt by General Electric. These defects were easily benched out. With the majority of the OD defects removed, the remaining ID defects were easier to identify in a second ultrasonic inspection. ID defects which exceeded the specification requirements were removed from the tube by sectioning. The metallographic appearance of typical ID defects of this type are shown in Figure 1.

The products made from the fourth and fifth T-111 alloy ingots (No. 111-D-1102 and 111-D-1765, respectively) were given a final vacuum anneal at 3000°F for one hour at the Boeing Company, Seattle, Washington. The final vendor inspection and testing of the products made from the fourth and fifth T-111 alloy ingots is in progress and a major portion of this material has been received. General Electric SPPS quality assurance inspection and testing of this received material is in progress and portions of the material have been released for manufacture.

The sixth T-111 alloy ingot was extruded at Canton Drop Forging Company on October 21, 1966. The extrusion parameters are presented in Table I. Rod rolling of the sixth T-111 alloy billet was completed the week of December



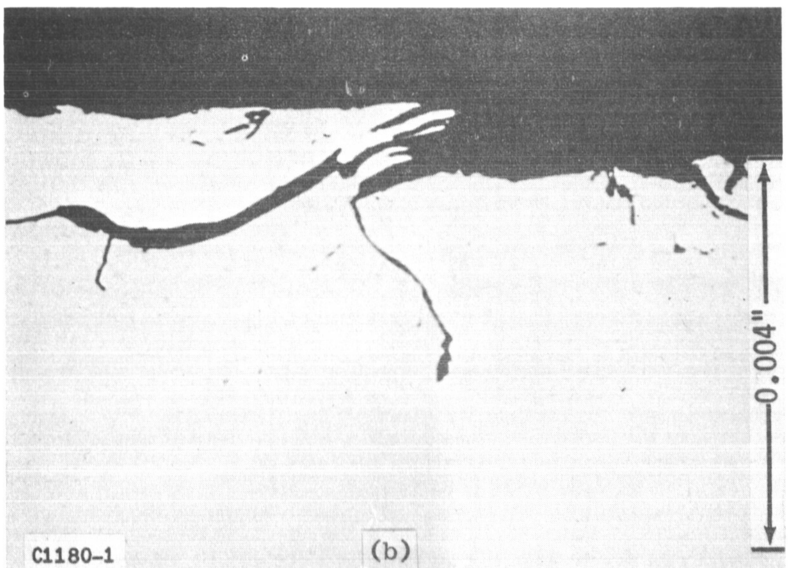


Figure 1. Photomicrographs of Typical Defects on the ID Surface and Inner Wall of 0.375-Inch OD x 0.065-Inch Wall T-111 Alloy Tubing.

a) Etchant: None 500X, C980223
b) Etchant: None 500X, C980222

TABLE I. EXTRUSION PARAMETERS FOR THE 6.72-INCH DIAMETER MACHINED T-111 ALLOY INGOT*

Machined Ingot Size - 6.72-inch diameter

Ingot Nose Geometry - 90° angle

Can Size - 7-1/4 inch OD x 1/4-inch thick wall Type 304SS seamless pipe with a molybdenum foil liner, a 2-inch thick Type 304SS nose block and 1/2-inch thick Type 304SS back-up block contained within the can with argon purge tubes attached.

Leader Block-Follow-up Block - 7-1/6-inch diameter x 6-inch long mild steel.

Container Size - 7.51-inch ID

Die Size/Design - 4.5-inch ID/conical

Die Coating - None

Extrusion Ratio - 2.7/1

Lubricant - Hot die grease similar to Fiske 604

Furnace Temperature/Soak Time in Salt Bath - 2200°F/2 hours

Extrusion Pressure (Hydraulic) - 1800 psi peak, average
1600 psi, runout, average

Maximum Allowable Pressure - Approximately 3850 psi

Cooling Procedure - Air cooled

Extrusion Direction of Ingot - Top of ingot was extruded through die first with hot top removed.

---

* Extruded at Canton Drop Forging Company, Canton, Ohio, on October 21, 1966.

5, 1966, at Braeburn Alloy Steel. The T-111 alloy rods have been rough machined into hollows and are now awaiting final vacuum annealing at Wolverine Tube. Finish machining will be done following the final anneal. The products to be obtained from this ingot are for back-up purposes and are not critical to initiating the loop fabrication phase of this program.

2. Cb-132M Alloy

A heat treatment of one hour at 2600°F has been selected for the 2-inch diameter Cb-132M alloy rod. A recently completed study has shown that this heat treatment results in improved room temperature ductility without appreciable recrystallization or reduction in the yield strength of the Cb-132M alloy.

The Cb-132M alloy as supplied by Universal Cyclops had been heat treated for one hour at 2400°F and had a room temperature ductility somewhat less than desirable for machining and utilization in Corrosion Loop I. It was anticipated that a slightly higher temperature heat treatment would result in increased carbide precipitation and reduce the amount of carbon in solid solution which would improve the room temperature ductility without an appreciable loss in yield strength. Approximately 0.25-inch thick slices were cut from the end of the Cb-132M alloy rod and subsequently sectioned into pie-shaped specimens. After chemical cleaning, the specimens were heat treated for one hour at 2500°, 2600°, 2700°, 2800°, and 2900°F at a pressure of less than 1×10^{-5} torr and then quenched in helium.

The microstructures and microhardnesses of the Cb-132M alloy before and after the one-hour heat treatments are shown in Figure 2. The first signs of slight recrystallization in the severely worked regions of the bar were observed in the specimen heat treated for one hour at 2700°F. Significant agglomeration of the carbide precipitate was observed after a heat treatment of one hour at 2900°F, Figures 3 through 5. The microhardness data indicate a rapid decrease in hardness after a one-hour heat treatment at 2700°F. From the microhardness and metallographic data, a temperature of less than 2700°F was suggested for a one-hour heat treatment.

The effects of longer time, lower temperature heat treatments were evaluated by heat treating a sample for 50 hours at 2500°F. The microstructure, shown

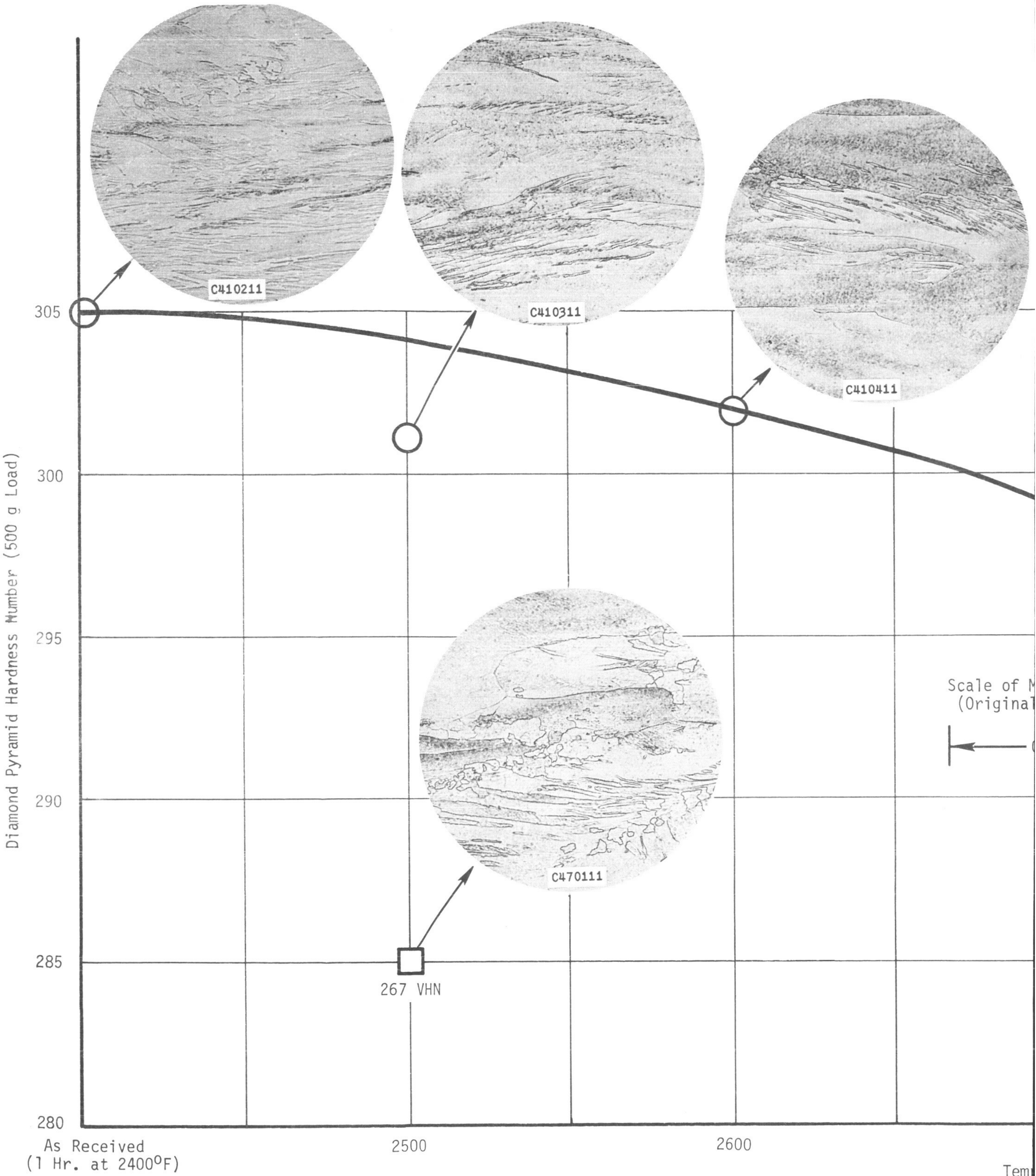
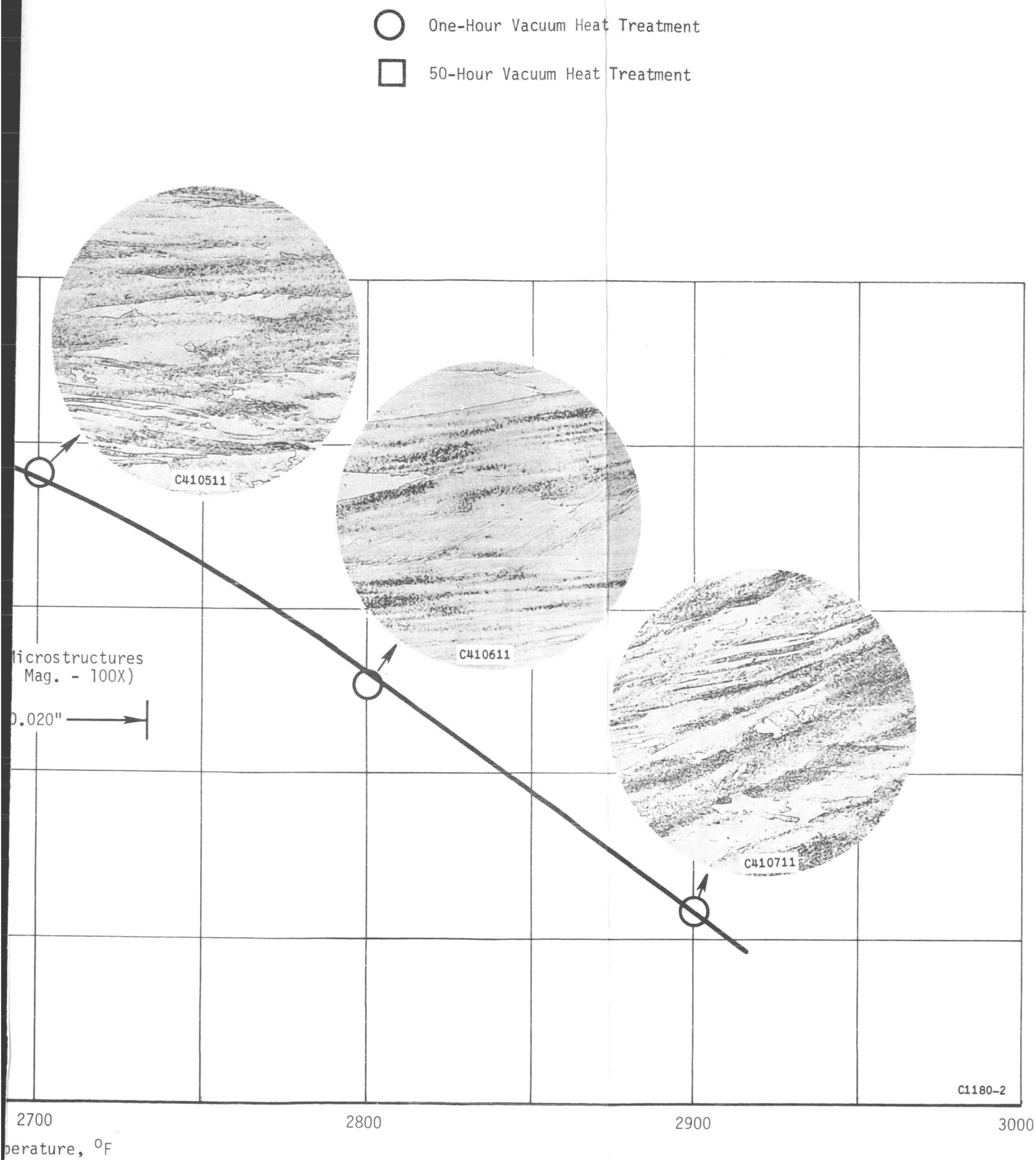


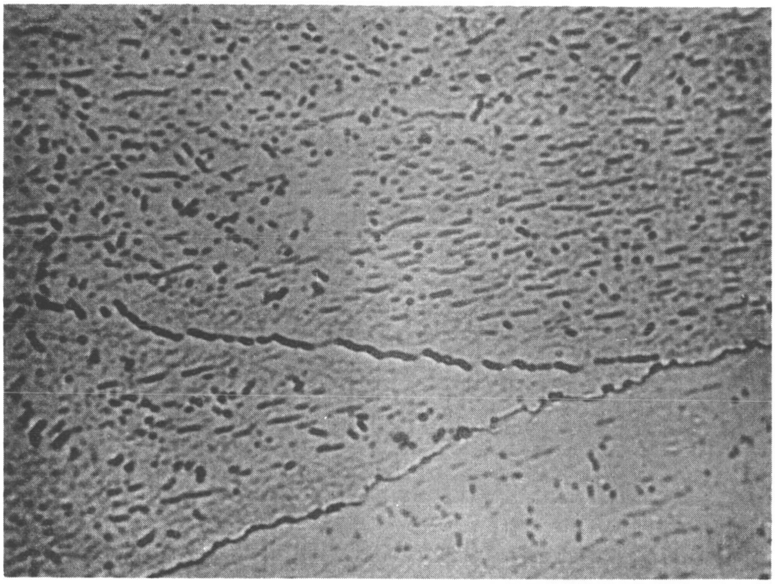
Figure 2. Microhardness and Mic
From 2.0-Inch Diamete

Etchant: 20mlHNO_3-20

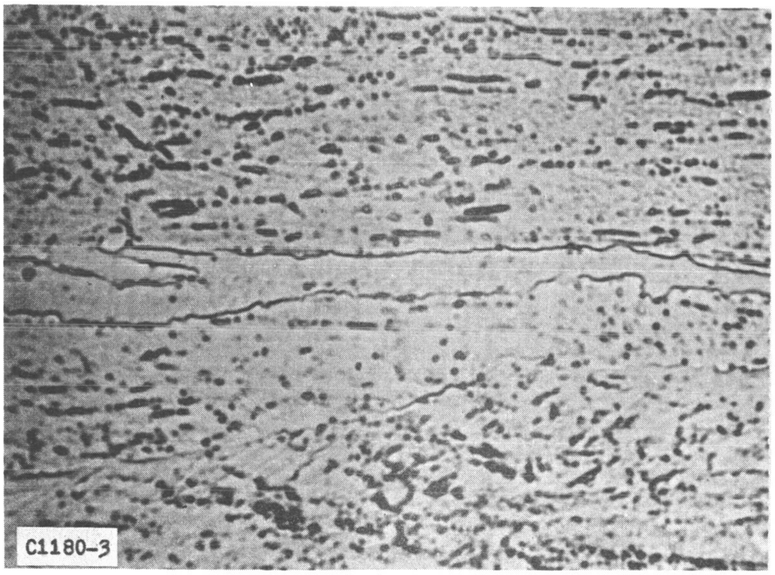


rostructure of Longitudinal Specimens Cut
r Cb-132M Alloy Rod.

mlHF-60mlGlycerine

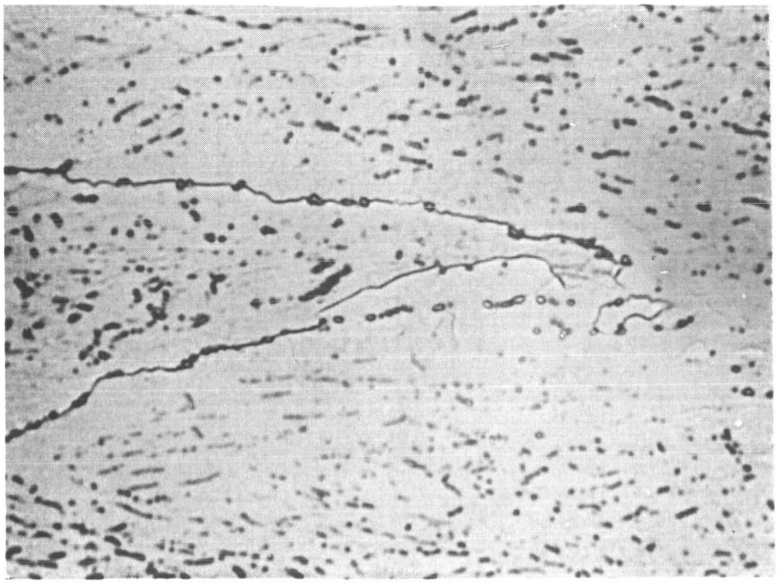


a) As-Received (Annealed 1 Hour at 2400°F)

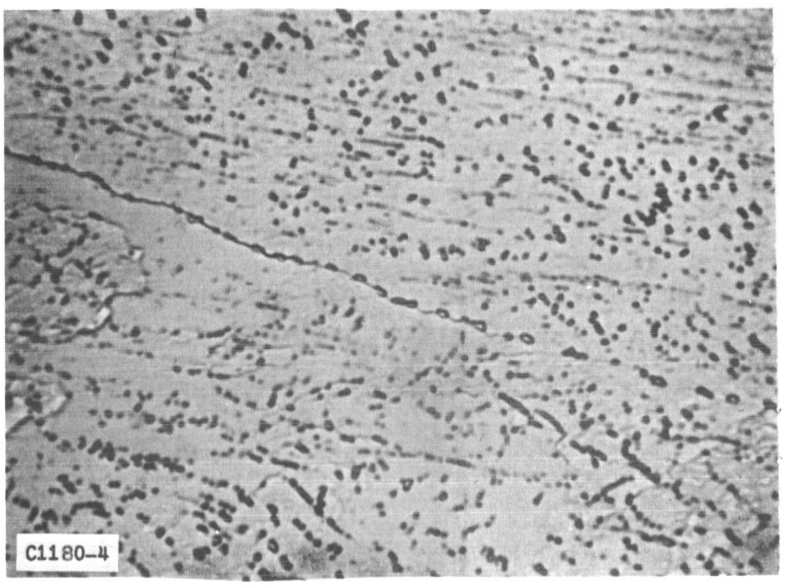


b) Annealed 1 Hour at 2500°F

Figure 3. Longitudinal Microstructure of 2.0-Inch Diameter Cb-132M Alloy Rod.
Etchant: 60mlGlycerine-20mlHNO_3-20mlHF Mag.: 2000X
a) C410212; b) C410313

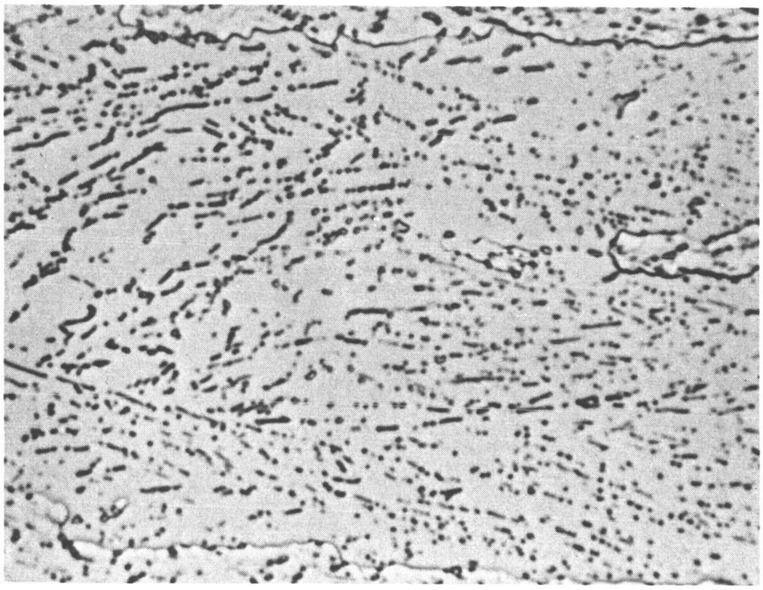


a) Annealed 1 Hour at 2600°F

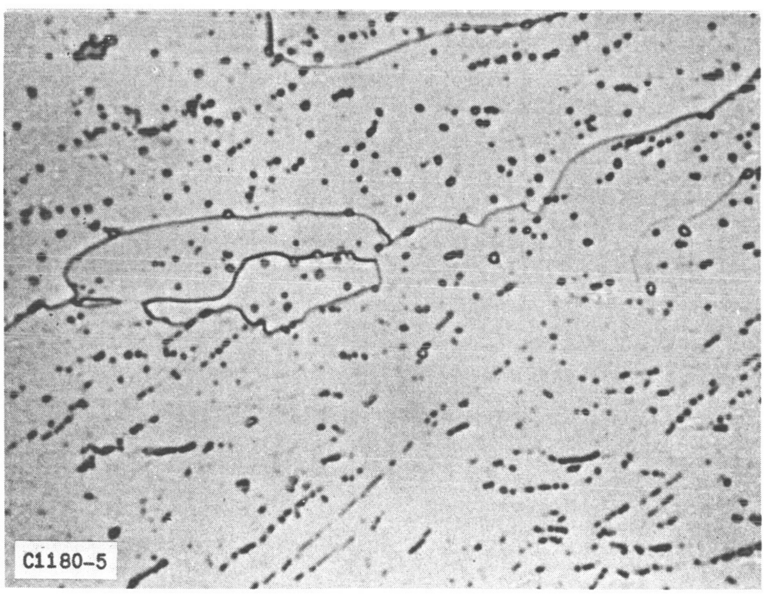


b) Annealed 1 Hour at 2700°F

Figure 4. Longitudinal Microstructure of 2.0-Inch Diameter Cb-132M Alloy Rod.
Etchant: 60mlGlycerine-20mlHNO_3-20mlHF Mag.: 2000X
a) C410413; b) C410512



a) Annealed 1 Hour at 2800°F



b) Annealed 1 Hour at 2900°F

Figure 5. Longitudinal Microstructure of 2.0-Inch Diameter Cb-132M Alloy Rod.
Etchant: 60mlGlycerine-20mlHNO_3-20mlHF Mag.: 2000X
a) C410613; b) C410712

in Figures 2 and 6 indicates significant recrystallization and agglomeration of the carbide precipitate. These changes are reflected in the low microhardness, Figure 2.

The final heat treatment selection was determined by evaluating the tensile behavior of specimens heat treated for one hour at 2500°F and one hour at 2600°F. Buttonhead tensile specimens, Figure 7 were machined from the 2-inch diameter Cb-132M rod such that the longitudinal axes of the rod and specimens were parallel. The finished tensile specimens were chemically cleaned and carefully inspected for surface defects using fluorescent penetrant techniques. Three tensile specimens were vacuum heat treated at pressures less than 1 x 10^{-5} torr for one hour at 2500°F, three for one hour at 2600°F, and the remaining specimens were left in the as-received condition (one hour at 2400°F). The tensile tests were conducted in air at room temperature to 225°F and were performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." A strain rate of 0.005 inch/inch/minute up to 0.6% offset and then 0.050 inch/inch/minute to fracture was used; the yield strength was determined by the 0.02% and 0.2% offset methods. The results of these tests are shown in Table II along with the tensile data supplied by Universal Cyclops Steel Corporation.

Reasonable ductility was observed in the as-received Cb-132M alloy material (one hour at 2400°F) at a tensile test temperature of 225°F. Both thermal heat treatments (one hour at 2500°F and one hour at 2600°F) resulted in good room temperature ductility, and the specimens which were heat treated at 2600°F exhibited slightly higher ductility than those heat treated at 2500°F. In all cases, no significant reductions in yield strength were observed. From these results and the earlier microstructure and microhardness results, a thermal heat treatment of one hour at 2600°F was selected for the 2-inch diameter Cb-132M alloy rod. Subsequently, the 2-inch diameter Cb-132M alloy rod was wrapped in new Cb-1Zr alloy foil and vacuum annealed for one hour at 2600°F. The 1-inch diameter rod, which had satisfactory room temperature ductility in the as-received condition (2400°F/1 hour), and 2-inch diameter Cb-132M alloy rod have been released for manufacture.

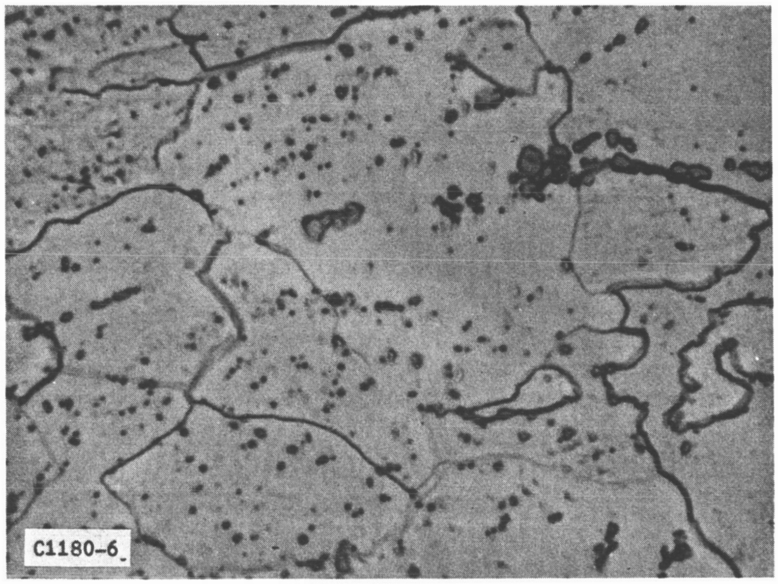


Figure 6. Longitudinal Microstructure of 2.0-Inch Diameter Cb-132M Alloy Rod Annealed 50 Hours at 2500°F.

Etchant: 60mlGlycerine-20mlHNO_3-20mlHF Mag.: 2000X

C470112

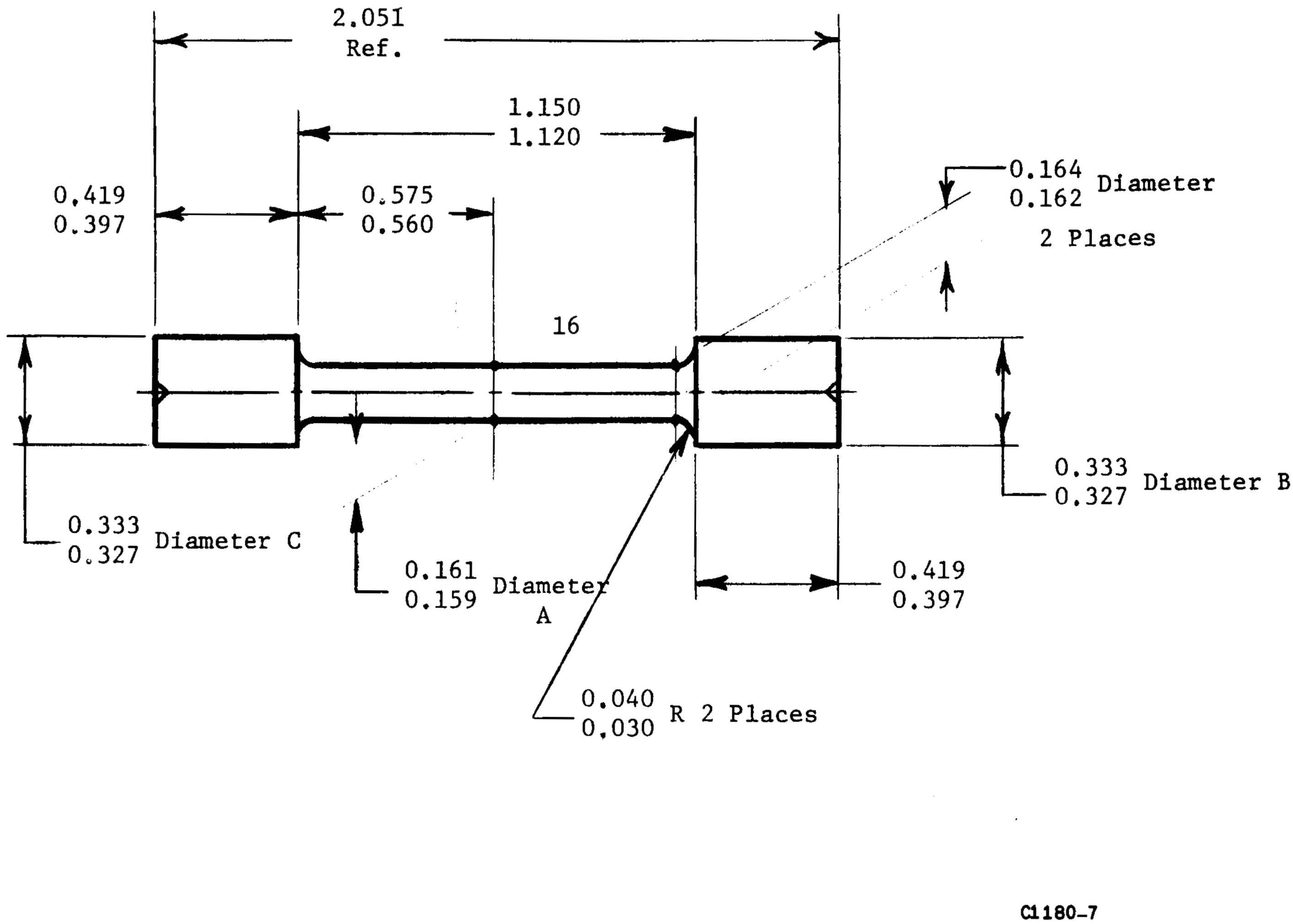


Figure 7. Design of Buttonhead Tensile Specimens Machined From Cb-132M Alloy and Mo-TZC Alloy Materials.

TABLE II. TENSILE PROPERTIES OF 2-INCH DIAMETER Cb-132M ALLOY ROD[(a)]

Specimen Number	Test Temp. °F	Ultimate Tensile Strength psi x 10^{-3}	0.02% Yield Strength[(b)] psi x 10^{-3}	0.2% Yield Strength[(b)] psi x 10^{-3}	Reduction in Area (%)	Elong. % in One Inch
AS-RECEIVED CONDITION (1 HOUR AT 2400°F)						
1[(c)]	R.T.	130.0	Not Reported	116.5	3.0	2.0
2[(c)]	R.T.	132.0	Not Reported	116.4	1.5	2.0
3[(d)]	150	123.0	106.0	112.0	4.0	3.0
4[(d)]	150	132.0	109.0	114.0	4.7	3.0
5	225	126.0	101.0	107.0	36.0	15.0
HEAT TREATED ONE HOUR AT 2500°F						
6	150	127.0	102.0	109.0	33.0	14.0
7	R.T.	132.0	108.0	115.0	15.0	11.0
8	R.T.	134.0	110.0	114.0	27.0	14.0
HEAT TREATED ONE HOUR AT 2600°F						
9	150	123.0	100.0	106.0	44.0	17.0
10	R.T.	132.0	109.0	114.0	44.0	17.0
11	R.T.	132.0	108.0	114.0	20.0[(e)]	13.0

(a) Longitudinal direction.

(b) Strain Rate: 0.005 inch/inch/minute up to 0.2% offset and then 0.050 inch/inch/minute to fracture.

(c) Data reported by Universal Cyclops Steel Corp.

(d) Failed at radius - minimum reduction in area at Center.

(e) Not at location of fracture.

3. Mo-TZC Alloy

A heat treatment study similar to that described in the previous section was performed on the Mo-TZC alloy mill products. The Mo-TZC alloy mill products were of various shapes and manufactured by two vendors (General Electric-LMCD and Climax Molybdenum Company using different working schedules, therefore, a single optimum heat treatment for all the Mo-TZC alloy mill products could not be selected. Results of this study resulted in the following courses of action:

a. The 0.75-inch thick plate (General Electric-LMCD) and the 2.0-inch diameter rod (Climax) were heat treated for 50 hours at 2600°F.

b. The 1.375-inch thick plate (General Electric-LMCD) will not be used in the Corrosion Loop I (T-111) Program because of its nonuniform microstructure.

c. The 1-inch diameter rod (Climax) will be utilized in the as-received condition (1 hour at 2400°F).

The heat treatment study was conducted with approximately 0.25-inch thick slices cut from the ends of each of the Mo-TZC alloy mill shapes. The slices from the plate material were cut into cubes and the slices from the rods were cut into pie-shaped specimens. After chemical cleaning, the specimens were heat treated for one hour at 2600°, 2700°, 2800°, 3000°, 3200°, 3400°, and 3750°F in a vacuum of less than 1 x 10^{-5} torr and subsequently quenched in helium.

The microstructures and microhardnesses of the 0.75-inch thick Mo-TZC alloy before and after the one-hour heat treatment are shown in Figure 8. Significant recrystallization was observed in the specimen heat treated for one hour at 2600°F as well as a low hardness. The data obtained for this particular heat treatment are considered anomalous with respect to the data obtained after performing other heat treatments on this material. The one-hour heat treatment at 2800°F is considered to be the condition in which significant recrystallization will occur in the 0.75-inch thick plate. Also, the microhardness data indicate a rapid decrease in hardness after a one-hour

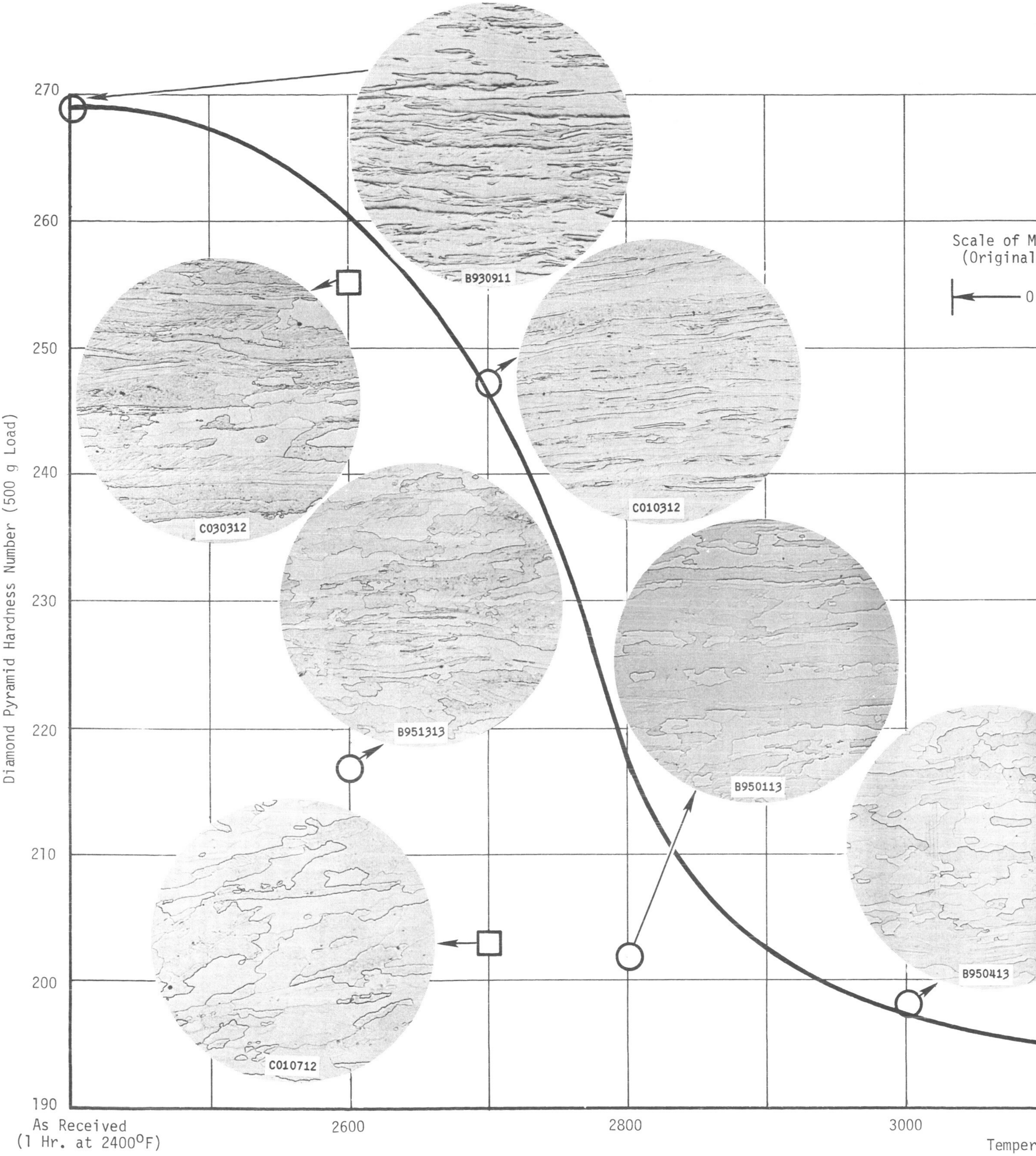
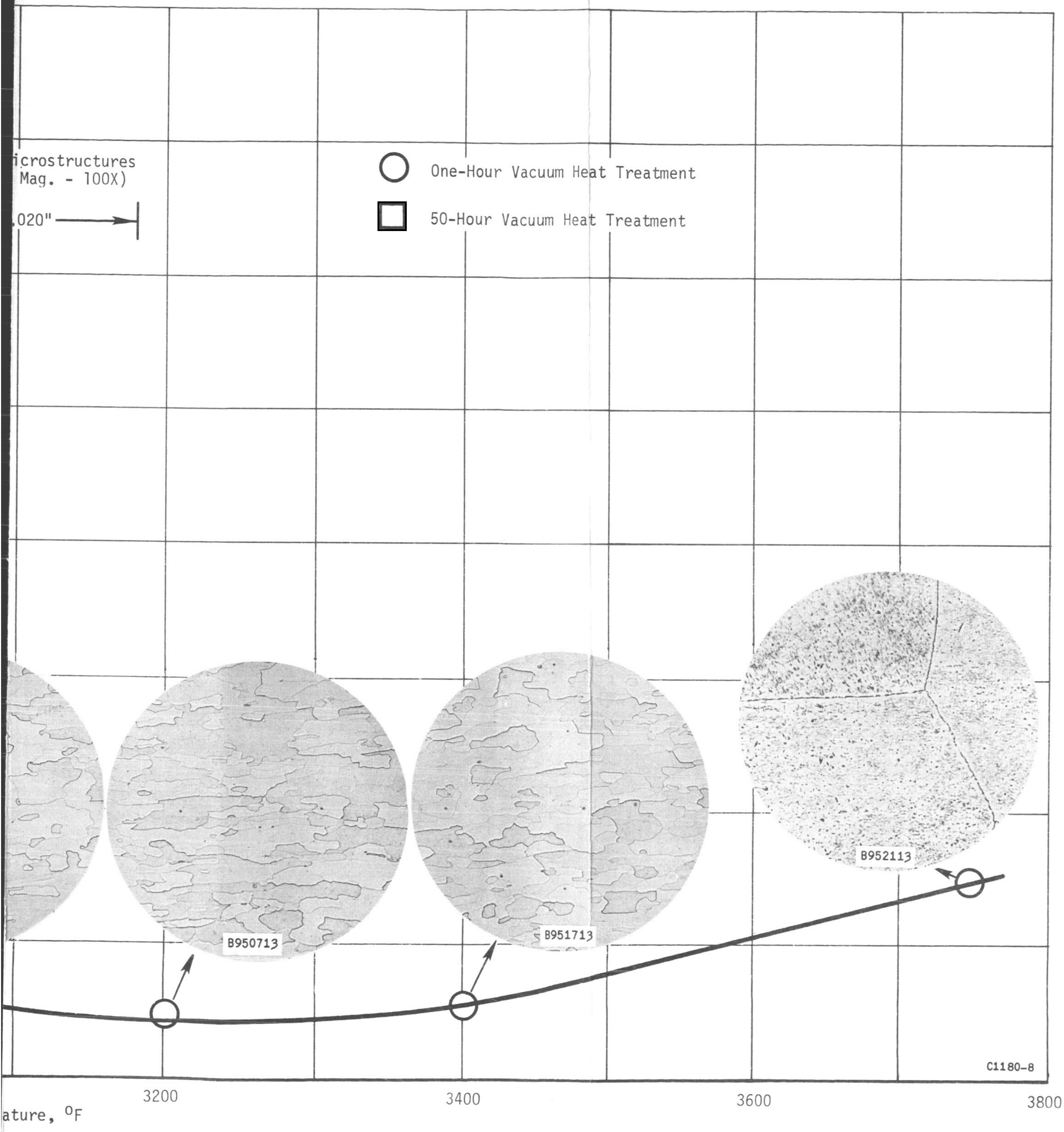


Figure 8. Microhardness and Microstruc
From 0.75-Inch Thick Mo-TZC
Treatment Temperature.

Etchant: 50% Murakamis



ture of Longitudinal Specimens Cut
Alloy Plate as a Function of Heat

heat treatment at a temperature of 2700°F. From the metallographic and microhardness data, a temperature of 2700°F or less was suggested for a one-hour heat treatment of the 0.75-inch thick plate.

The effects of longer time heat treatments on microstructure and hardness of the 0.75-inch thick plate were evaluated by heat treating samples for 50 hours at 2600° and 2700°F. The microstructures, Figure 8, indicate only a slight amount of recrystallization in the specimen heat treated at 2600°F for 50 hours and a significant amount of recrystallization in the specimen heat treated at 2700°F for 50 hours. The microhardness data, Figure 8, are in agreement with the microstructures in that the hardness of the specimen heat treated at 2700°F was considerably reduced. These results indicate that a 50-hour heat treatment at 2600°F also may be beneficial in achieving greater tensile ductility at the lower temperatures. A longer time heat treatment has the advantage of allowing more time for carbon precipitation.

The microstructures and microhardness of the 1.375-inch thick Mo-TZC alloy plate before and after the one-hour heat treatments are shown in Figure 9. Significant recrystallization was observed after heat treating for one hour at 3200°F. This high recrystallization temperature is attributed to the relatively small amount of cold work that this material received during processing, as shown in the as-received microstructure which is noticeably less uniform than the microstructure of the other materials. In general, the microstructure data for the one-hour heat treatments below 3200°F indicate that recovery is the major process taking place with very small localized areas, which received a greater amount of effective cold work, starting to recrystallize. Since the slope of the hardness curve is fairly uniform up to 3200°F and microstructural changes are slight, it would be desirable to maintain the temperature of a one-hour heat treatment for the 1.375-inch thick Mo-TZC alloy as low as possible to reduce degradation of strength and high enough to precipitate carbon from solution in a reasonable time period. The effects of longer time heat treatments on the 1.375-inch thick plate were also evaluated by heat treating samples for 50 hours at 2600° and 2700°F. The microstructures of these specimens are shown in Figure 9 and indicate that recovery is predominate with some recrystallization in small localized areas in the specimen heat treated for 50 hours at

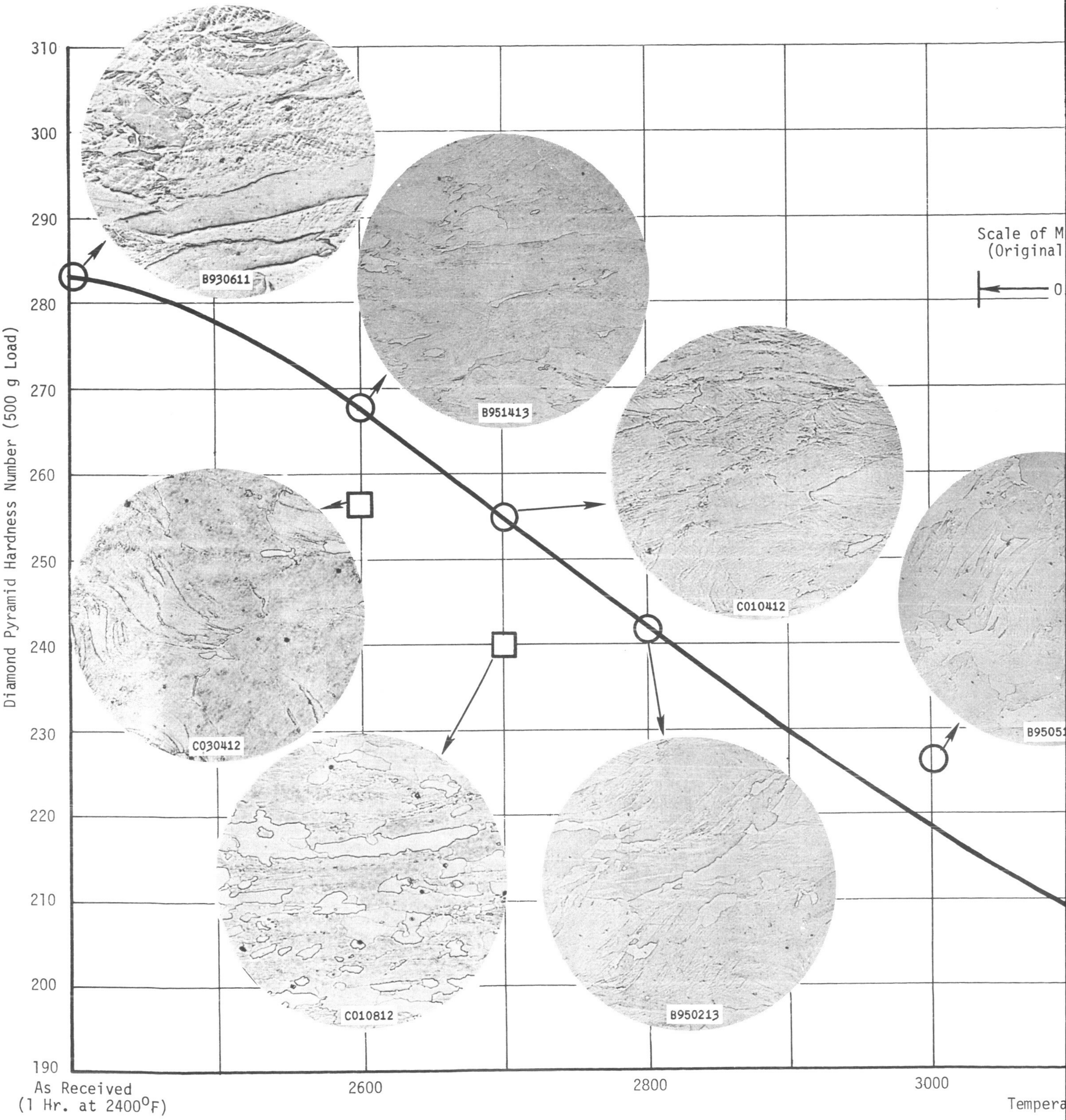
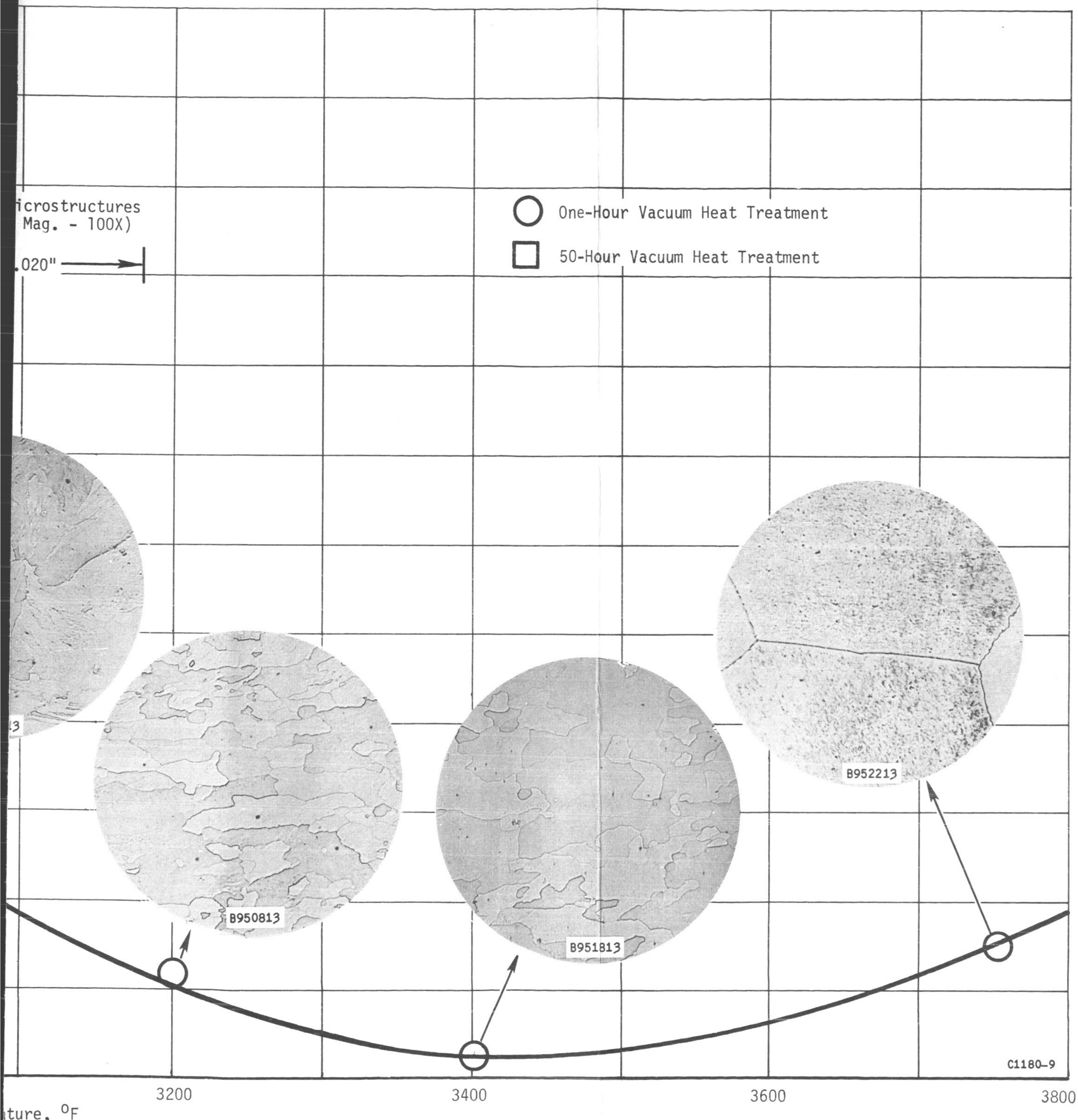


Figure 9. Microhardness and Microstr
From 1.375-Inch Thick Mo-T
Treatment Temperature.
Etchant: 50% Murakamis



ructure of Longitudinal Specimens Cut
TZC Alloy Plate as a Function of Heat

2600°F and a significant amount of recrystallization in the specimen heat treated at 2700°F for 50 hours. A decrease in hardness was observed in both specimens, Figure 9, with the hardness values being slightly less than the one-hour heat treatments at 2600° and 2700°F. These data indicate that a 50-hour heat treatment at 2600°F would be the preferred heat treatment to improve the low temperature tensile ductility of the 1.375-inch thick plate with a minimal reduction in yield strength.

The room temperature tensile ductility of the 1.0-inch diameter Mo-TZC alloy rod produced by Climax Molybdenum is satisfactory for use in the as-received condition (2400°F/1 hour). However, heat treatment studies were conducted with this material to determine its recrystallization characteristics. The microstructures and microhardnesses after the one-hour heat treatments are shown in Figure 10. Significant recrystallization was observed in the specimen which was heat treated at 3000°F for one hour. The microhardness data show a rapid decrease in hardness after one hour at 2800°F. The effects of longer time heat treatments (50 hours at 2600° and 2700°F) on the 1.0-inch diameter material are shown in Figure 10. Metallographic examination revealed the initiation of recrystallization in both heat treatments but to an insignificant amount. A slight decrease in microhardness also was observed.

The microstructures and microhardnesses of the 2.0-inch diameter Mo-TZC alloy rod produced by Climax Molybdenum before and after the one-hour heat treatments are shown in Figure 11. Significant recrystallization was observed after heat treating for one hour at 3000°F and a slight decrease in microhardness was noted after the one-hour heat treatment at 2800°F. These results indicate that a one-hour heat treatment to precipitate carbon from solution should be carried out at a temperature of 2800°F or less. The effects of longer time heat treatments (50 hours at 2600° and 2700°F) on the 2.0-inch diameter material are shown in Figure 11. Significant recrystallization was observed in the specimen heat treated at 2700°F and is reflected in the low microhardness of the specimen. The specimen heat treated at 2600°F for 50 hours exhibited a very small amount of recrystallization and a slight decrease in hardness. These data indicate that a 50-hour exposure at 2600°F could be used for the final heat treatment of the 2.0-inch diameter material.

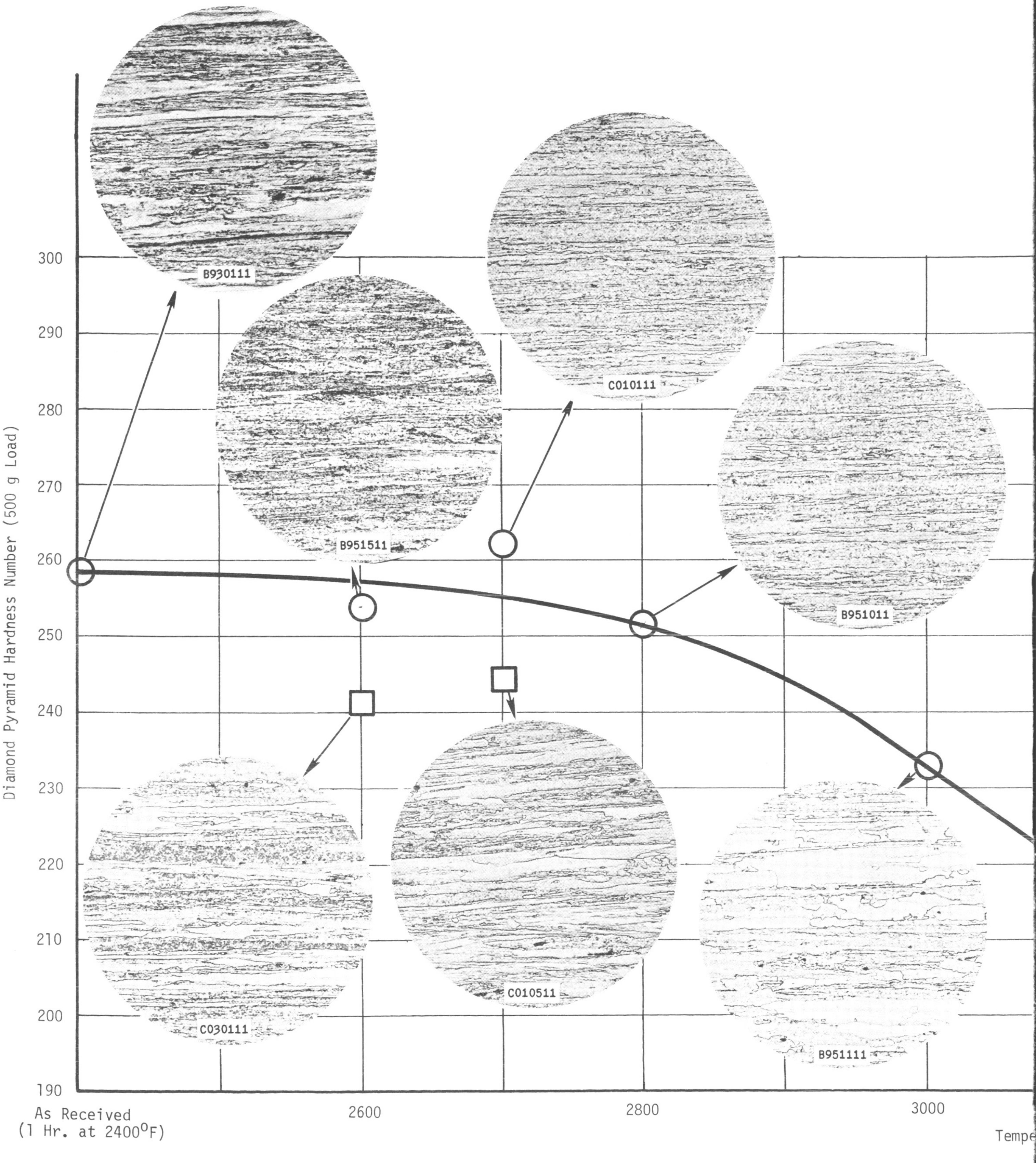
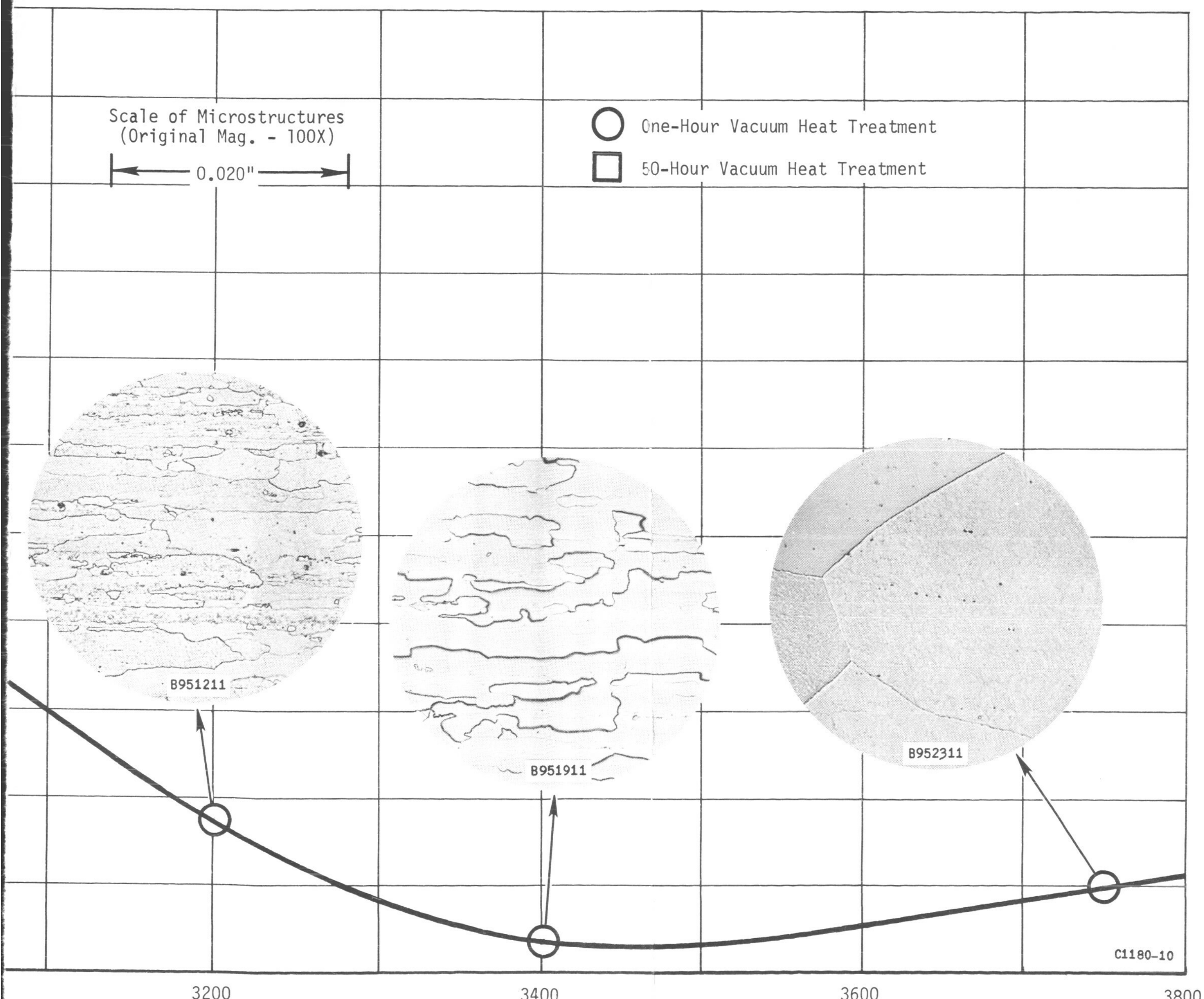


Figure 10. Microhardness and Micro
From 1.0-Inch Diameter
Treatment Temperature.

Etchant: 50% Murakamis



erature, °F

structure of Longitudinal Specimens Cut
Mo-TZC Alloy Rod as a Function of Heat

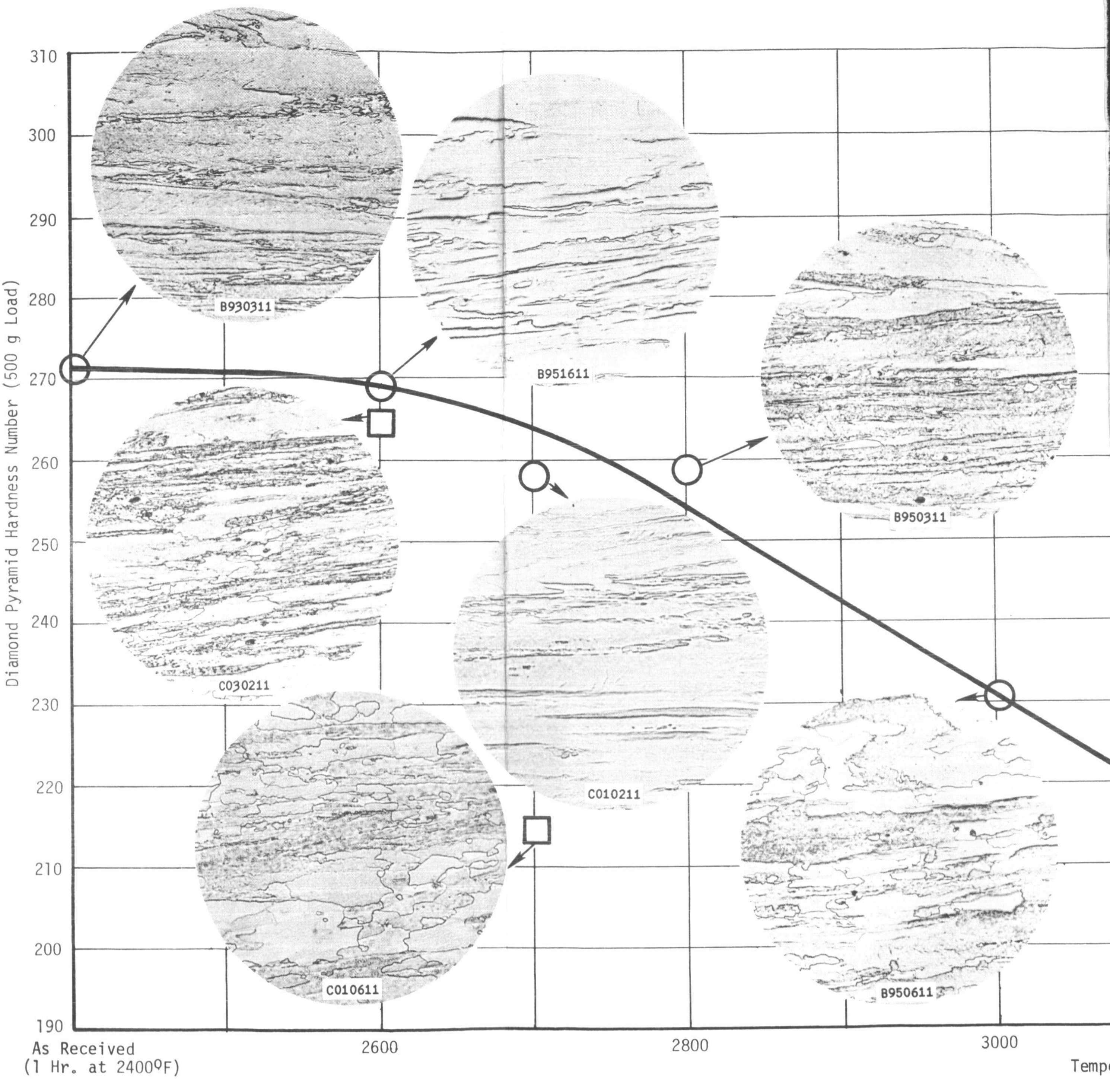
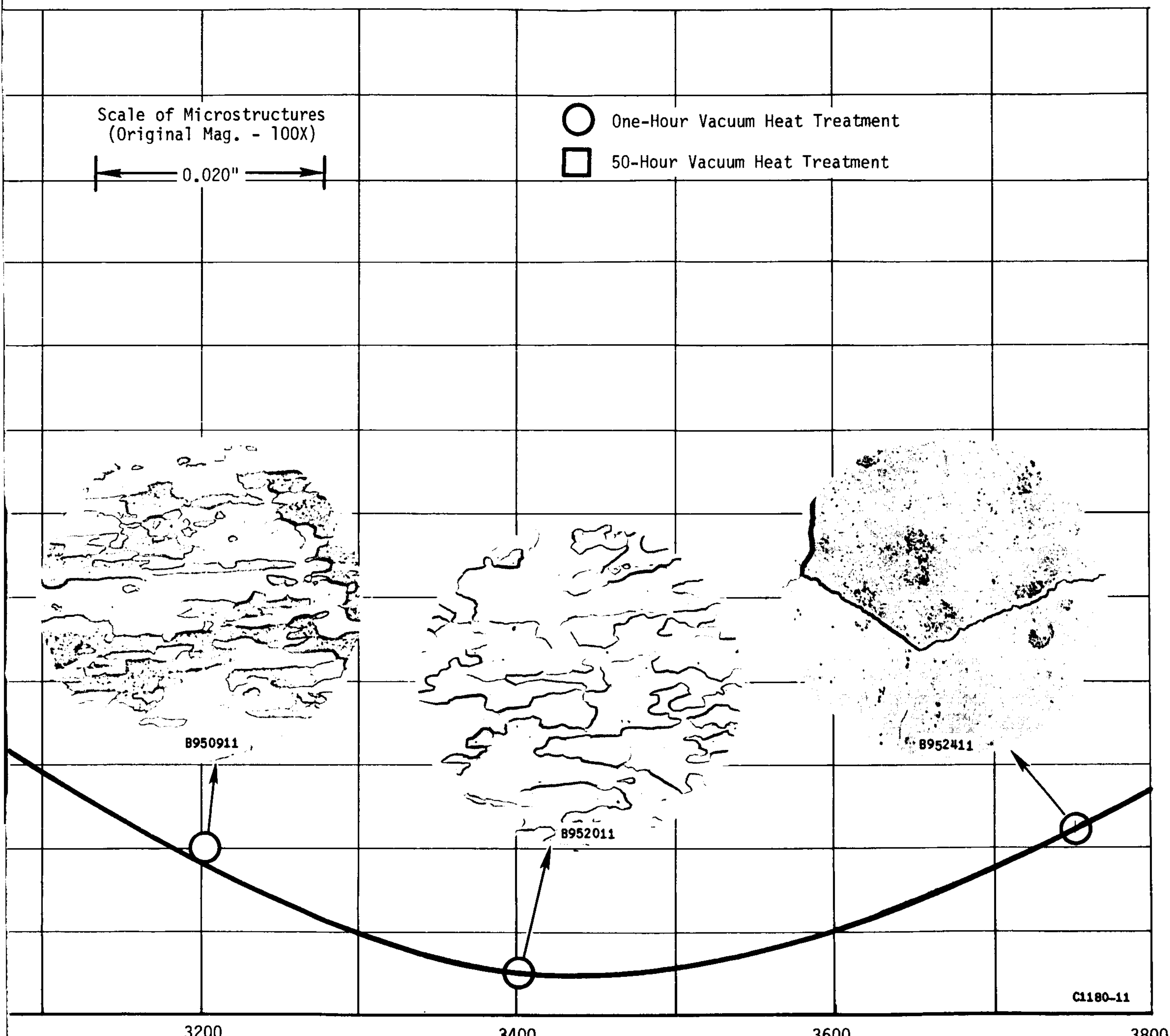


Figure 11. Microhardness and M
From 2.0-Inch Diame
Treatment Temperatu

Etchant: 50% Murak



ature, °F

crostructure of Longitudinal Specimens Cut er Mo-TZC Alloy Rod as a Function of Heat e.

mis

As a result of these studies, 50 hours at 2600°F was selected as a promising heat treatment for the comparison of tensile properties with the material in the as-received condition of one hour at 2400°F. The tensile testing was done in the same manner as that described in the Cb-132M alloy heat treatment study described above, except that test temperatures ranged from room temperature to 400°F. The results of these tests along with the data reported by the vendors are shown in Table III.

In general, an increase in room temperature ductility and a decrease in room temperature yield strength was observed in the specimens tested from all four mill shapes which were heat treated for 50 hours at 2600°F. It was concluded that the improved ductility was more significant to the utilization of these materials in Corrosion Test Loop I (T-111) than the observed decrease in strength except for the 1.0-inch diameter rod material. The 1.0-inch diameter Mo-TZC alloy rod exhibited very good ductility in the as-received condition (heat treated one hour at 2400°F) and the slight increase observed after heat treating for 50 hours at 2600°F was considered insignificant. Therefore, no additional heat treatment was selected for the 1.0-inch diameter rod and it will be used in Corrosion Test Loop I (T-111) in the as-received condition.

The room temperature ductility achieved in the 0.75-inch thick plate after heat treating for 50 hours at 2600°F was considered acceptable; therefore, this heat treatment was utilized for the 0.75-inch thick Mo-TZC alloy plate.

Since the machining of the 1.375-inch thick plate and 2.0-inch diameter rod will be done by electric discharge machining techniques, which can be done readily at temperatures on the order of 150°F, the ductility achieved in both the 1.375-inch thick plate and the 2.0-inch diameter rod at 150°F after heat treating 50 hours at 2600°F is considered acceptable. However, even though good ductility was achieved in the 1.375-inch thick plate, it was decided that the non-uniform microstructure observed in the metallographic studies made it undesirable to use this material in Corrosion Test Loop I (T-111). The 2.0-inch diameter rod will be used after heat treating at 2600°F for 50 hours.

TABLE III. TENSILE PROPERTIES FOR Mo-TZC ALLOY PLATE[a] AND ROD[b]

Material	Specimen No.[b]	Test Temp. °F	Ultimate Tensile Strength psi x 10^{-3}	0.02% Yield Strength[c] psi x 10^{-3}	0.2% Yield Strength[c] psi x 10^{-3}	Reduction in Area (%)	Elong. % in 1 Inch
0.75-Inch Thick Plate	(d)	R.T.	111.3	Not Reported	101.9	--	2.1
	2	150	111.0	72.0	86.0	11.5	9.5
	1	225	107.0	72.5	82.5	11.0[e]	8.0
	3 (f)	78	106.0	67.1	75.0	9.0	7.5
	4 (f)	150	88.1	53.3	61.0	28.5	21.0
1.375-Inch Thick Plate	(d)	R.T.	94.7	Not Reported	92.8	--	0.3
	(d)	R.T.	96.6	Not Reported	95.2	--	0.4
	21	225	90.0	65.2	73.2	2.5	3.0
	25	300	99.0	73.0	80.0	4.0	3.5
	22	400	88.7	62.4	70.9	51.5	15.5
	23 (f)	81	96.5	70.3	80.0	1.0[g]	3.5
	24 (f)	150	98.5	62.0	72.3	12.0	12.0
1.0-Inch Diameter Rod	(d)	R.T.	118.4	--	106.0	--	15.5
	(d)	R.T.	118.0	--	109.1	--	32.5
	29	88	114.0	81.0	97.8	53.5	25.0
	30	88	117.2	82.8	101.0	38.0	20.0
	31 (f)	80	107.0	80.8	89.0	38.0[h]	26.0
2.0-Inch Diameter Rod	(d)	R.T.	104.1	--	96.1	--	1.5
	(d)	R.T.	106.1	--	97.1	--	2.0
	10	82	99.0	80.5	94.7	1.5	1.0
	11	84	102.0	85.8	95.8	1.0	1.0
	12	150	97.0	75.0	86.0	2.0	1.5
	13	150	98.5	72.0	84.9	1.0	1.0
	15	225	110.0	73.7	82.3	47.5	15.5
	9	225	103.0	72.7	83.5	50.0	17.5
	14	300	98.5	74.0	83.6	54.5	15.5
	16 (f)	78	107.0	72.5	84.3	5.0	3.5
	17 (f)	150	101.0	69.2	78.4	12.0	13.0

Footnotes on next page.

TABLE III. (Cont'd)

(a) Longitudinal to final rolling direction.

(b) All specimens unless otherwise marked are from as-received material which was annealed for 1 hour at 2400°F.

(c) Strain Rate: 0.005 inch/inch/minute to 0.2% yield strength.

(d) Data reported by vendor.

(e) RA reported was at the location of fracture; the maximum was 37.5%.

(f) Specimen annealed 50 hours at 2600°F at a pressure of $<1 \times 10^{-5}$ torr.

(g) RA reported was at the location of fracture; the maximum RA was 3.8%.

(h) RA reported was at the location of fracture; the maximum RA was 72.0%.

The 0.75-inch thick Mo-TZC alloy plate and the 2.0-inch diameter Mo-TZC alloy rod were chemically cleaned, wrapped in fresh Cb-1Zr alloy foil, and subsequently heat treated at 2600°F for 50 hours in a vacuum of $< 1 \times 10^{-5}$ torr. All of the Mo-TZC alloy material needed for use in the Corrosion Test Loop I (T-111) has been released for manufacture.

B. ALKALI METAL PURIFICATION

The lithium hot trap shown in Figure 12 prior to assembly was fabricated and installed on the dolly with the vacuum-argon system. After outgassing the hot trap at temperatures up to 1500°F, 28.5 lbs of lithium were introduced into the hot trap. After operating the hot trap for 126 hours at 1500°F, the gettering action of the zirconium foil had reduced the nitrogen concentration of the lithium from 700-800 ppm to less than 10 ppm. The oxygen concentration was not changed by the gettering operation and remained at 150 ppm. The carbon concentration previously reduced from 150 ppm to 100 ppm by filtration through a five-micron stainless steel filter was further lowered to 30 ppm by the hot trapping operation.

The lithium still, shown in Figure 13, was completed and attached to the hot trap and vacuum-argon system. The still is currently being outgassed, at room temperature, while the thermocouples and heating elements are being attached. Subsequently, the still will be outgassed at the distillation temperature conditions until a room temperature outgassing rate of less than one micron-liter per hour is obtained.

C. FABRICATION

The fabrication status of T-111 Corrosion Loop critical components is as follows:

1. Slack Diaphragm Transducers - The 0.005-inch thick T-111 alloy diaphragms have been formed. The body flanges are being machined; however, delivery has been delayed until the end of January. Bimetallic joints between Cb-1Zr and Type 316SS required for the NaK side of the transducer were received with incomplete inspection reports and have been returned to the vendor. Subsequently, it was determined that rework was required and delivery is now scheduled for the end of January.

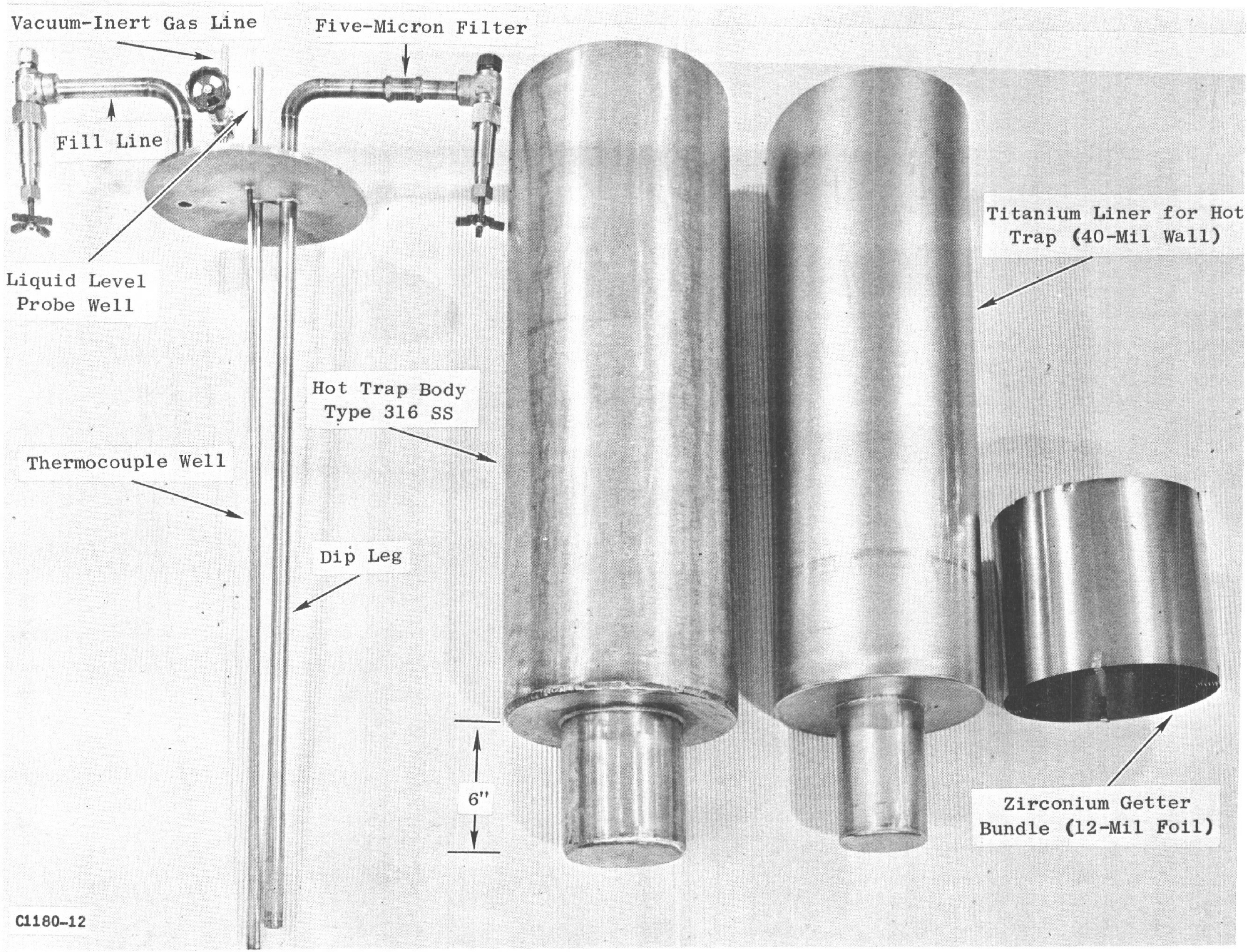


Figure 12. Hot Trap for Purifying Lithium for Corrosion Loop I (T-111) Prior to Assembly. (C66060312)

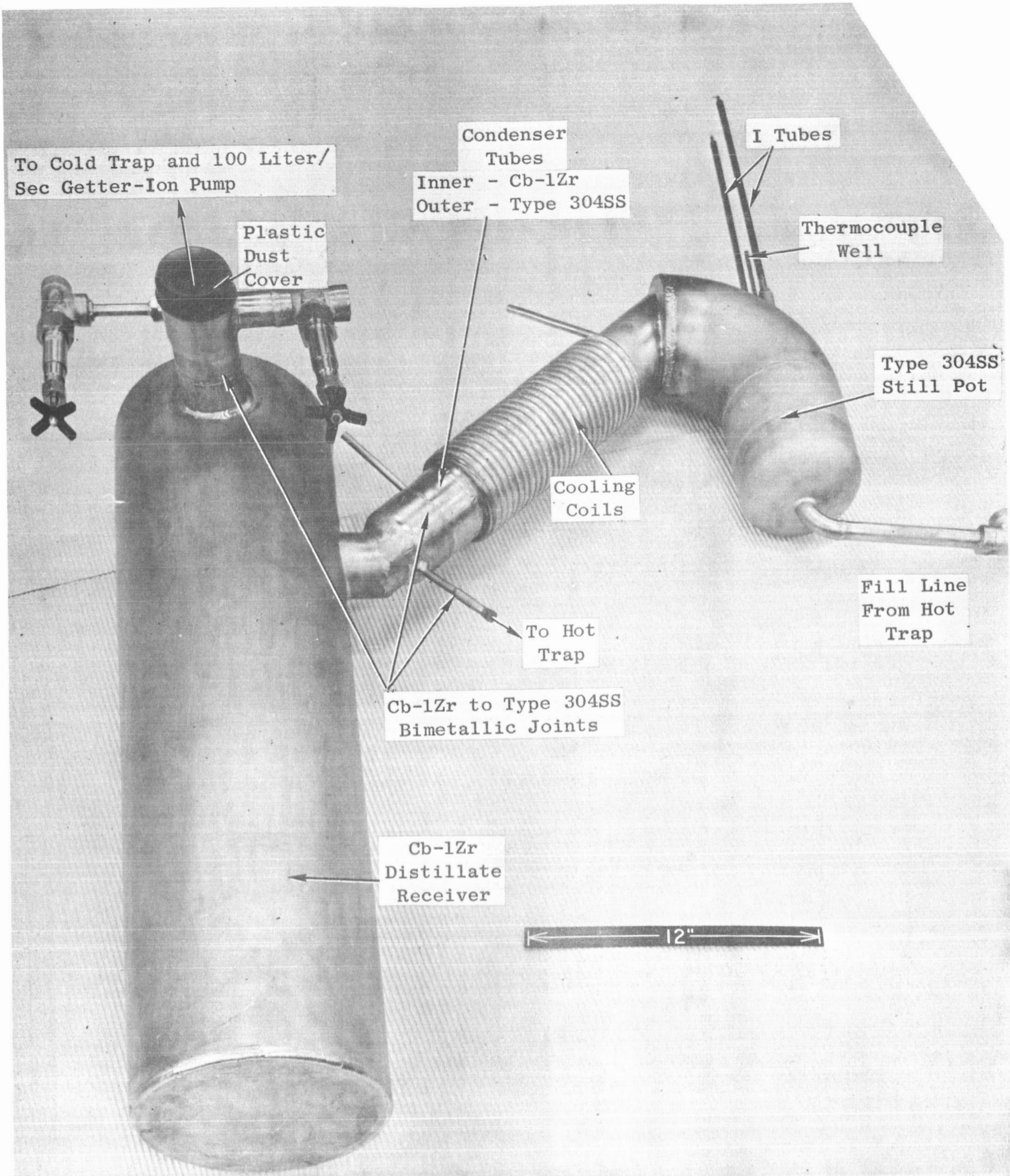


Figure 13. Assembled Lithium Still for Corrosion Loop I (T-111).
(Orig. C66121230)

2. Lithium and Potassium EM Pump Ducts - Both EM pump duct outer wrappers were being machined with delivery expected by the end of December. However, the loss of the key machining vendor to this program has forced redirection to other potential vendors. Delivery is now expected near the middle of February. The potassium helical duct has been machined. It is anticipated that the additional T-111 alloy required for the lithium pump duct will be released for manufacture by the end of January.

3. Throttle and Isolation Valves - The vendor, Hoke, Inc., has scheduled a delivery date of March 24, 1967 for machined parts. The Cb-1Zr bellows for these valves were shipped from GE-SPPS to Hoke, Inc. on January 20, 1967.

4. Turbine Simulator - Delivery of the Mo-TZC and Cb-132M alloy blades have been delayed because of cracking observed in the first three blades produced from GE Mo-TZC alloy. The vendor is currently re-evaluating the machining sequence in cooperation with GE-SPPS. The order for nozzle machining has been placed with delivery of completed nozzle assemblies expected by the end of February. The 2-1/4-inch OD x 3/8-inch wall T-111 alloy tube for the turbine simulator casing is scheduled for release for manufacture by the end of January.

5. Condenser. A 1-inch x 2-inch x 32-inch long T-111 alloy bar has been committed for evaluation of drilling procedures for the center hole. The first trial was unsuccessful due to breakage of the carbide drill. Other potential sources for deep hole drilling are currently being evaluated. It is anticipated that new drilling trials will be initiated by the end of January.

6. Boiler - Machined components are on order with delivery scheduled for mid-January, 1967.

Machining orders for the remaining loop components have been placed with deliveries scheduled for mid-January, 1967.

D. QUALITY ASSURANCE

1. T-111 to Cb-1Zr Alloy Welding

Metallographic examination of T-111 to Cb-1Zr weld specimens provided a plausible explanation for the differences in hardness and bend ductility previously described in Quarterly Progress Report No. 3[(1)].

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(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 4 for Period Ending 4-15-66, NASA Contract NAS 3-6474, NASA-CR-72027, pp 24-27.

Representative photomicrographs (weld zone and Cb-1Zr heat affected zone) of the T-111 to Cb-1Zr automatic welds in selected heat treat conditions are shown in Figure 14. The lower hardness of the overaged specimens was associated with significant homogenization in the fusion zone and random precipitation of second phase particles in the Cb-1Zr heat affected zone. No obvious microstructural changes were noted in the heat affected zone of the T-111 alloy as a result of the different heat treatments.

Microstructure comparisons of the manual weld samples after the various heat treatments shown in Figure 15 revealed the probable cause for the superior stability of those welds prepared with Cb-1Zr filler. The 2300°F for one hour and 2400°F for one hour heat treatments were capable of significant homogenization of the fusion zones of these welds, while the same thermal cycles apparently had little effect on microstructures of the welds prepared with T-111 filler. Also, the higher welding temperatures associated with the use of T-111 filler may have caused more relative solutioning of the base Cb-1Zr into the fusion zone leading to depressed aging reaction rates and apparent lesser stability in that area.

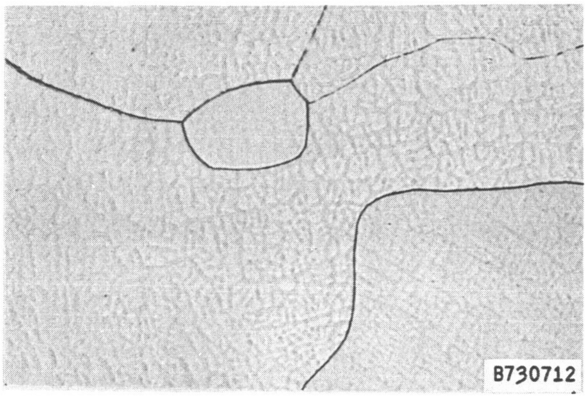
Small particles of T-111 alloy, as exemplified in Figure 16, were also detected in the fusion zones of welds prepared with T-111 alloy filler. Their presence may have resulted from the following:

1. Incomplete melting of the filler metal because the lowest possible weld power was used to minimize melting distortion of the Cb-1Zr member.
2. Partial melting of the T-111 base metal caused by momentary contact of the welding arc.

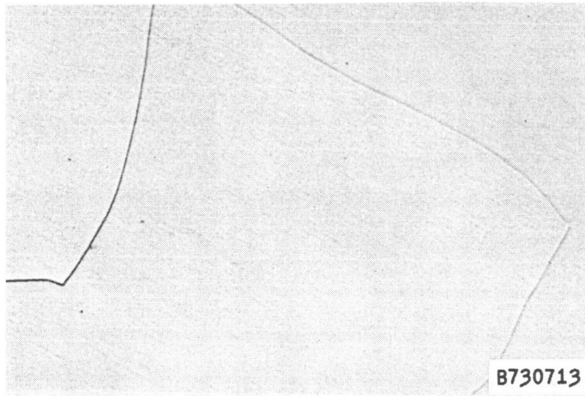
These particles could act as stress concentrations during bending thereby lowering the ductility of the weld.

The data obtained from bend testing, hardness testing and microstructural examination of the T-111 to Cb-1Zr welds indicated that optimum stability of manual welds could be obtained through the use of Cb-1Zr filler material and postweld annealing treatments in the 2300° and 2400°F temperature range.

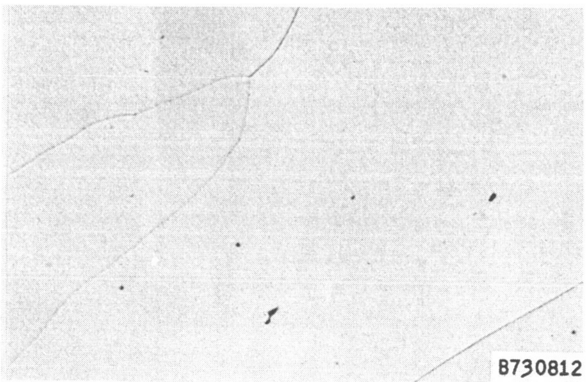
Weld Zone	Heat Affected Zone (Cb-1Zr)



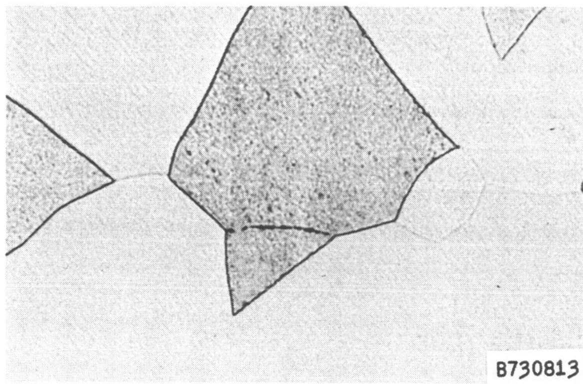
1500°F/50 Hr.



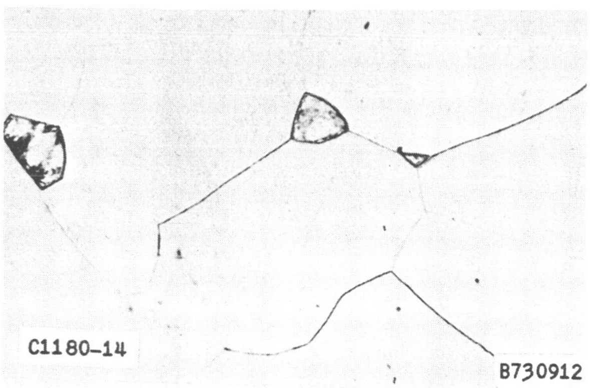
1500°F/50 Hr.



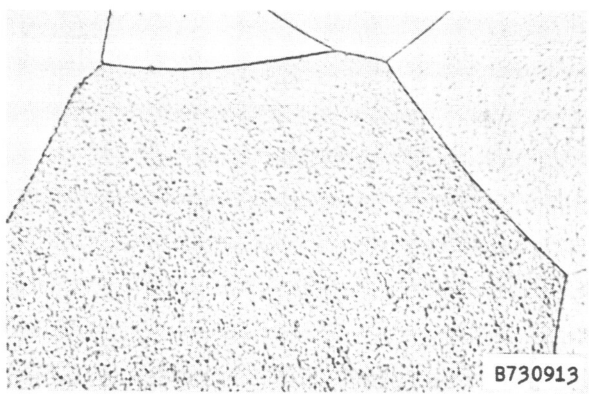
2300°F/1 Hr.



2300°F/1 Hr.



2400°F/1 Hr.

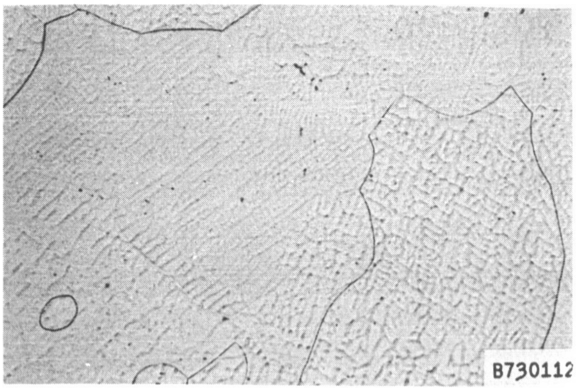


2400°F/1 Hr.

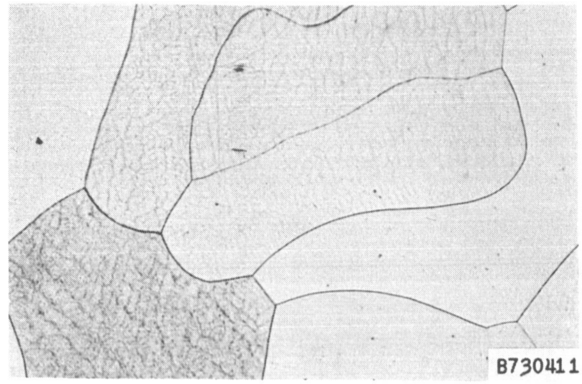
Figure 14. Microstructures of Automatic Weldments of Cb-1Zr to T-111 (0.040-Inch Thick Sheet) Prepared Without Filler Additions Following Various Postweld Heat Treatments.

Etchant: $30gmNH_4F-50mlHNO_3-20mlH_2O$ Mag.: 250X

T-111 Filler | Cb-1Zr Filler

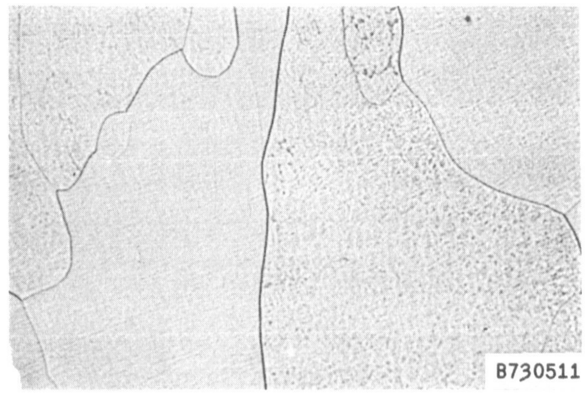


1500°F/50 Hr.



1500°F/50 Hr.

2300°F/1 Hr. + 1500°F/50 Hr.



2300°F/ 1 Hr. + 1500°F/50 Hr.

2400°F/1 Hr. + 1500°F/50 Hr.

2400°F/1 Hr. + 1500°F/50 Hr.

Figure 15. Microstructures of Manual Weldments of Cb-1Zr to T-111 (0.080-Inch Thick Plate) Prepared With Cb-1Zr and T-111 Filler Material Following Various Postweld Heat Treatments.

Etchant: 30gmHN_4F-50mlHNO_3-20mlH_2O Mag.: 250X

Figure 16. Cb-1Zr to T-111 Weldments Prepared With T-111 Filler Showing T-111 Particle Which was Washed Into the Fusion Zone. Specimen was Heat Treated at 1500°F for 50 Hours Following Welding. (B730111)

Etchant: $30gmNH_4F-50mlHNO_3-20mlH_2O$ Mag.: 100X

E. ADVANCED TANTALUM ALLOY CAPSULE TESTS

Delivery of the ASTAR 811 and ASTAR 811CN alloys from Westinghouse Astronuclear Laboratory is expected by February 15, 1967. All other materials necessary for this program have been received.

The T-111 alloy capsule shells and outer downcomer tubes, which are necessary for lithium thermal convection capsule fabrication, were fabricated successfully from 0.040-inch sheet. Preparations have been made to electron beam seam weld these tubes. The necessary welding fixtures for the potassium reflux capsule fabrication have been designed and constructed. All the shielding, heaters, electrodes, and fixturing for the potassium reflux capsule test facility and lithium thermal convection capsule test facility have been fabricated.

IV. FUTURE PLANS

A. All the refractory alloy materials for Corrosion Loop I (T-111) will be inspected and released for manufacturing of loop components.

B. Fabrication of a majority of the loop components will be completed.

C. The lithium will be distilled.

D. The ASTAR 811 and ASTAR 811CN alloys will be received and specimens will be contaminated with oxygen as required. The checkout test will be initiated.

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PUBLISHED REPORTS

Quarterly Progress	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
Report No. 3 (NASA-CR-54911)	January 15, 1966
Report No. 4 (NASA-CR-72029)	April 15, 1966
Report No. 5 (NASA-CR-72057)	July 15, 1966
Report No. 6 (NASA-CR-72177)	October 15, 1967